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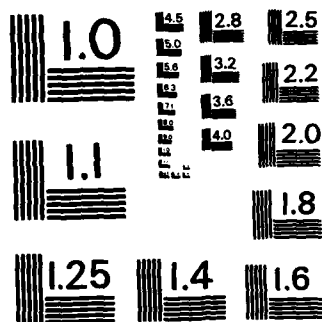
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TECHNICAL REPORT NO. 22

Magnetic and Electrical Properties of Rhodium(III)OXIDE(III)

by

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A

MAGNETIC AND ELECTRICAL PROPERTIES OF RHODIUM(III)OXIDE(III)

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ABSTRACT:

$\text{Rh}_2\text{O}_3(\text{III})$ crystallizes with an orthorhombic corundum-related structure and is Pauli paramagnetic. Its electrical behavior is characterized by a small activation energy, and the Seebeck coefficient indicates p-type conduction. This oxide may be considered as a semimetal, and its properties are discussed in terms of face and edge sharing of RhO_6 octahedra.

Introduction

The synthesis and crystallographic properties of the compounds belonging to the Rh-O system have been the subject of numerous studies (1-8). It has been found that three rhodium(III) oxides can exist under different conditions: the low temperature, low pressure form ($\text{Rh}_2\text{O}_3(\text{I})$); a high temperature, high pressure structure ($\text{Rh}_2\text{O}_3(\text{II})$); and a high temperature, ambient pressure compound ($\text{Rh}_2\text{O}_3(\text{III})$).

$\text{Rh}_2\text{O}_3(\text{I})$ has been obtained when rhodium compounds such as the hydrous oxide, nitrate, sulfate, or chloride are decomposed between 600-750°C (1-3). The resulting product crystallizes with the corundum structure, and when this phase is heated between 750-1000°C, it transforms irreversibly to $\text{Rh}_2\text{O}_3(\text{III})$, which is more thermally stable. The phase $\text{Rh}_2\text{O}_3(\text{III})$ has also been prepared by direct oxidation of rhodium metal (2,5). A major difficulty in obtaining pure samples of $\text{Rh}_2\text{O}_3(\text{III})$ is its tendency to dissociate at relatively low temperature. The decomposition temperature depends on the oxygen pressure, and decreases from 1130°C at 760 torr, to 900°C at 10 torr (3). In a reducing atmosphere, this oxide decomposes readily to rhodium metal at about 100-150 C°(3). Rhodium(III)oxide(III) crystallizes with an

orthorhombic corundum-related structure (space group $Pbca$) (5). $Rh_2O_3(II)$ has been obtained by heating rhodium oxide (produced by heating rhodium(III) chloride in air at 800°C) at 1200-1500°C and 65 kb pressure (6).

Although $Rh_2O_3(I)$ and $Rh_2O_3(III)$ are the most common forms of the rhodium(III) oxides, their magnetic and electronic properties have not been completely determined. Rhodium(III)oxide(II) has been reported to be a semiconductor with a room temperature resistivity of 130 $\Omega\text{-cm}$ and an activation energy of 0.16 (6), but the nature of this behavior has not been explained.

In this study, $Rh_2O_3(III)$ was prepared and characterized with regard to its crystallographic, magnetic, and electrical properties.

Synthesis

Rhodium(III)oxide(III) was prepared by the oxidation of finely divided rhodium metal (Engelhard Inc. 99.99%) in an oxygen atmosphere at 800°C until constant weight was obtained. A silica tube was used to contain the rhodium powder. The product was ground each day in an agate mortar, and then reheated. After the last heating, the compound was allowed to cool slowly to room temperature. The metal-to-oxygen ratio was determined by reduction of the final product at 900°C with a mixture of 85% argon - 15% hydrogen. The oxygen content obtained was 18.9 weight percent, which corresponds to the composition Rh_2O_3 .

Discs of $Rh_2O_3(III)$ were made by pressing aliquots of the polycrystalline material at 50 kb for 30 minutes at 650°C. Under these conditions, only $Rh_2O_3(III)$ exists, since $Rh_2O_3(II)$ requires higher temperatures and pressure to form. After pressing, the discs were cooled slowly, transferred to a silica tube, and annealed in air for six days at 600°C to ensure reoxidation of any rhodium metal which might have been formed during the sintering process. The discs were then examined by x-ray analysis in order to confirm the existence of pure, homogeneous $Rh_2O_3(III)$. No changes of the diffraction patterns were observed when compared to the starting polycrystalline $Rh_2O_3(III)$.

The density of these discs was determined using a hydrostatic technique based on Archimedes' principle (9). Perfluoro-1-methyldecalin (Pierce Chemical Co.) was used as the liquid medium, and its density was calibrated before each measurement with a high-purity silicon crystal ($D_{22^\circ C} = 2.328 \text{ g/cm}^3$). An average density of 7.80 (g/cm^3) was found for the discs, which is greater than 95% of the calculated density of $Rh_2O_3(III)$ (8.19 g/cm^3).

Magnetic and Electrical Properties

Magnetic measurements were performed using a Faraday balance (10). The magnetic susceptibility was measured as a function of temperature from liquid nitrogen to 300K at a field strength of 10.4 kOe. The data were then corrected for core diamagnetism ($-32 \times 10^{-4} \text{ emu/g } Rh_2O_3$) (11). The lack of any field dependency of the susceptibility showed the absence of ferromagnetic impurities.

The van der Pauw method (12) was used to measure the electrical resistivities. Contacts were made by the ultrasonic soldering of indium directly onto the sintered discs of $\text{Rh}_2\text{O}_3(\text{III})$, and their ohmic behaviors were established by measuring their current-voltage characteristics. The Seebeck coefficient was obtained by applying a temperature gradient to a sintered disc of $\text{Rh}_2\text{O}_3(\text{III})$ and recording the resulting voltage difference.

Results and Discussion

The crystal structure of $\text{Rh}_2\text{O}_3(\text{III})$ was first determined by Biesterbos and Hornstra (5). The structure projected along the orthorhombic x-axis is shown in Figure 1. All of the rhodium ions are octahedrally coordinated by oxygen ions, which in turn are bonded to four rhodium ions. It can be seen that pairs of $[\text{RhO}_6]$ octahedra share faces, and each pair is connected to another pair within the plane, and to pairs above and below the plane, by means of edge-sharing.

$\text{Rh}_2\text{O}_3(\text{III})$ prepared in this study was orthorhombic (space group Pbca), and the following cell constants were determined: $a = 5.146(3)\text{\AA}$, $b = 5.440(1)\text{\AA}$, and $c = 14.71(1)\text{\AA}$. These results are in good agreement with those reported previously for $\text{Rh}_2\text{O}_3(\text{III})$ (2,5).

FIG. 1

Unit cell in the structure of $\text{Rh}_2\text{O}_3(\text{III})$ as viewed along the orthorhombic x-axis.

Magnetic Properties

The magnetic data for $\text{Rh}_2\text{O}_3(\text{III})$ shown in Figure 2 indicate a weak temperature-independent paramagnetism (Pauli paramagnetic behavior) with $\chi = 150 \times 10^{-6} \text{emu/mole Rh}_2\text{O}_3$ ($0.59 \times 10^{-6} \text{emu/g Rh}_2\text{O}_3$). This behavior implies probable delocalization of electrons in Rh_2O_3 .

Electrical Properties

The temperature variation of the electrical resistivity of $\text{Rh}_2\text{O}_3(\text{III})$ is shown in Figure 3; the resistivity increases slightly from 40-cm at room temperature to 410-cm at liquid nitrogen temperature. Figure 3 shows a curvature and yields a maximum value for the activation energy of less than 0.05 . This indicates that $\text{Rh}_2\text{O}_3(\text{III})$ does not follow typical semiconductor behavior. A value of $-37\text{mV}/^\circ\text{C}$ was obtained for the Seebeck coefficient of $\text{Rh}_2\text{O}_3(\text{III})$. The magnitude of this result also suggests metallic behavior; the sign indicates predominantly p-type conduction.

FIG. 2

Variation of magnetic susceptibility with temperature for $\text{Rh}_2\text{O}_3(\text{III})$.

FIG. 3

Variation of resistivity with temperature for $\text{Rh}_2\text{O}_3(\text{III})$.

The magnetic and electrical behavior of $\text{Rh}_2\text{O}_3(\text{III})$ may be explained by assuming the existence of narrow d-bands and acceptor levels located slightly above them. Such bands probably form because each RhO_6 octahedra shares a face and edges with neighboring octahedra. Consequently, the t_{2g} orbitals of each rhodium ion are always directed to the nearest neighboring rhodium ions along these directions. In $\text{Rh}_2\text{O}_3(\text{III})$, the Rh-Rh distance across the shared face is $2.69 \pm 0.05 \text{ \AA}$ (5), which is similar to the equivalent distance reported for V_2O_5 . V_2O_5 crystallizes with the related corundum structure and possesses V-V bands formed by t_{2g} - t_{2g} interactions (13,14). Since metal-metal distances in these structures allow for the overlapping of 3d orbitals, it is expected that the same overlap may occur in $\text{Rh}_2\text{O}_3(\text{III})$, considering the larger extension of 4d wave functions. By symmetry considerations, any overlap of orbitals across shared edges may also have some oxygen character.

In $\text{Rh}_2\text{O}_3(\text{III})$ the existence of acceptor levels located above filled valence bands is consistent with the existence of $\text{Rh}(\text{IV})$ ions. The existence of such acceptor levels close to the valence band is consistent with the small observed activation energy obtained from Figure 3. Electrons from the filled valence band can easily be promoted to the acceptor levels, leaving behind holes which can give rise to the observed p-type conduction.

Conclusions

$\text{Rh}_2\text{O}_3(\text{III})$ has been prepared and crystallizes with an orthorhombic corundum-related structure. The building block of this framework is an RhO_6 octahedron, which can share a face and edges with neighboring octahedra. This type of bonding allows for the possibility of direct rhodium-rhodium or indirect rhodium-oxygen-rhodium interactions, and appears to determine the electrical and magnetic properties of the oxide. $\text{Rh}_2\text{O}_3(\text{III})$ shows Pauli paramagnetism, and its electrical behavior is characterized by a small activation energy. The Seebeck coefficient indicates p-type conduction. This behavior is consistent with the formation of a filled d-band, with rhodium or rhodium-oxygen character, and acceptor levels lying close to this band. These levels are due to the presence of a small amount of $\text{Rh}(\text{IV})$ in the $\text{Rh}_2\text{O}_3(\text{III})$.

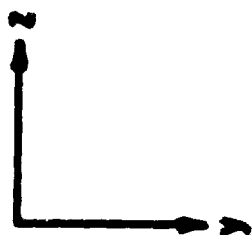
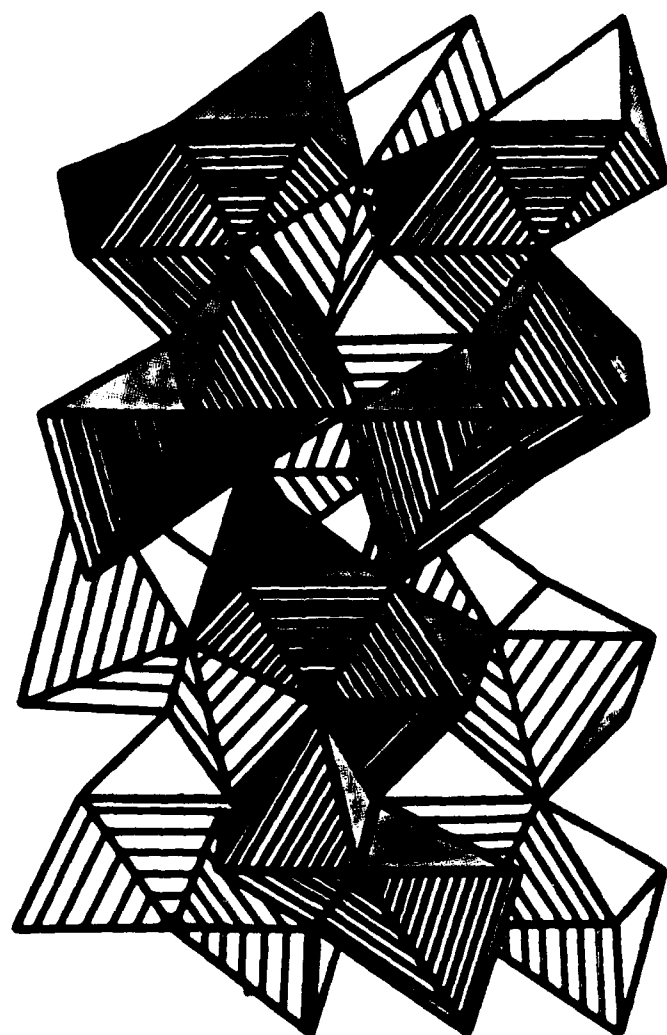
The determination of the magnetic properties as well as some of the electrical properties of $\text{Rh}_2\text{O}_3(\text{III})$ indicates that this oxide may be considered a semimetal.

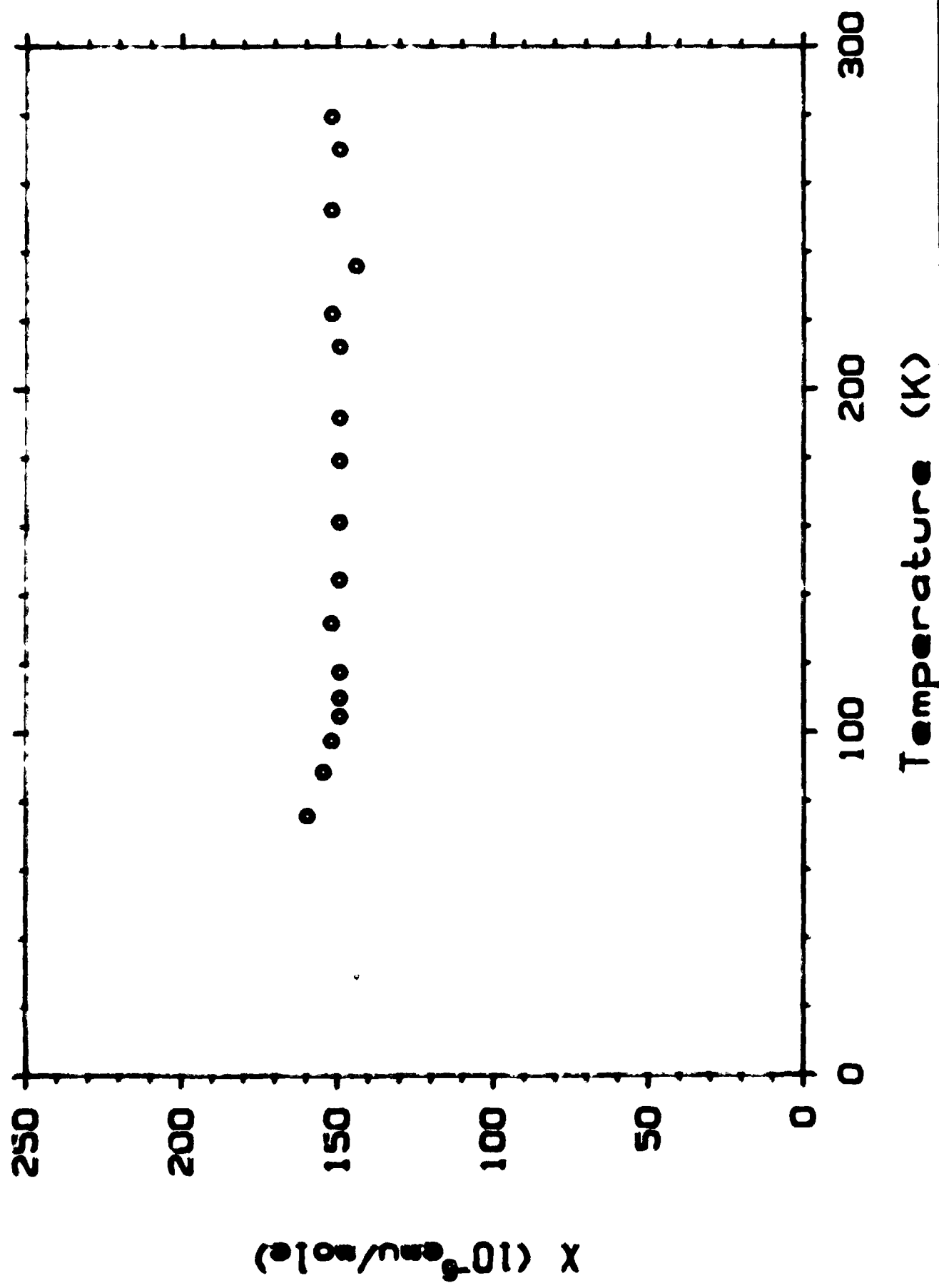
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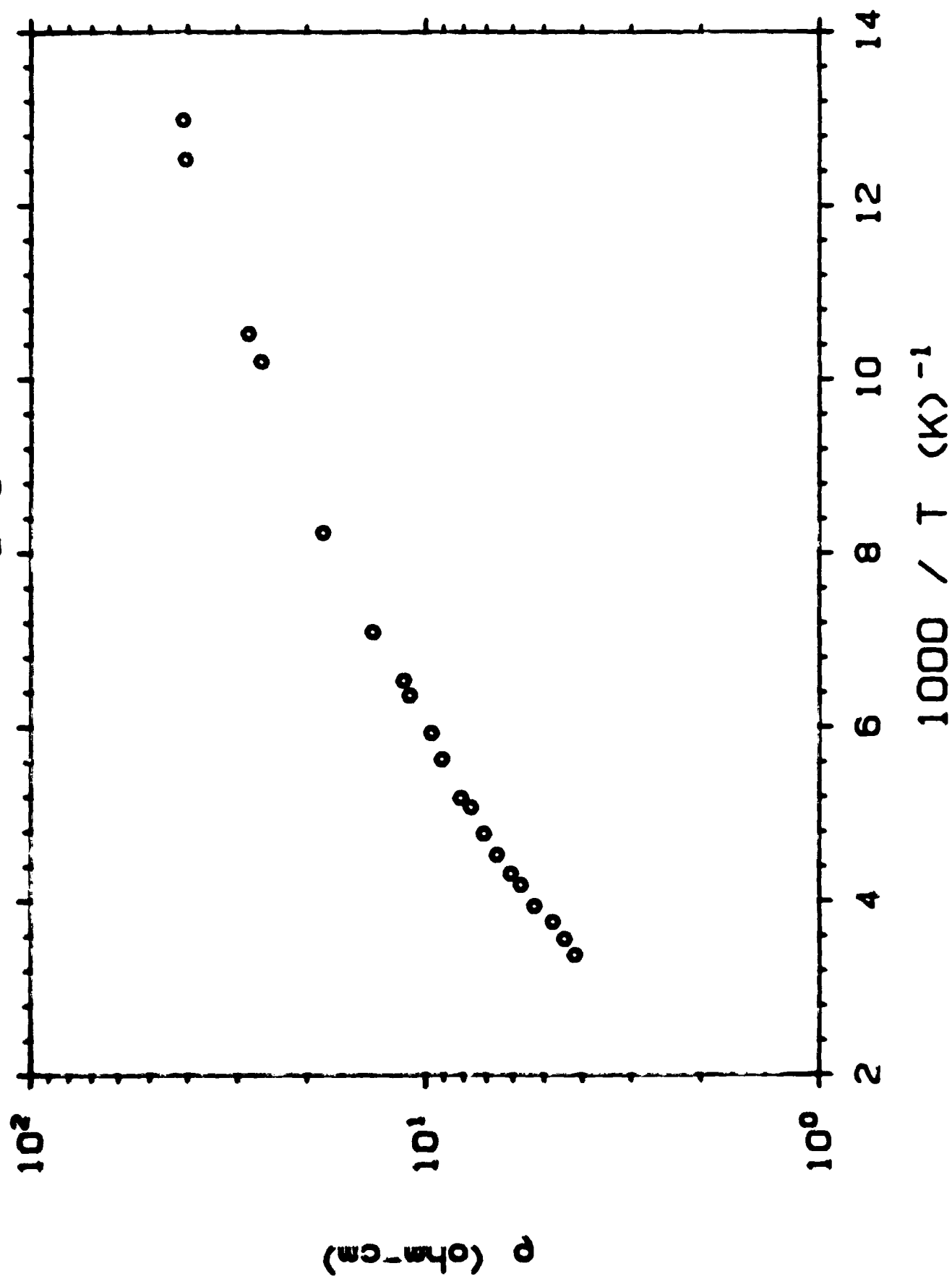
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Rh_2O_3



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